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To: Dr. D. Mills
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CARBON-14 CRYOGENIC DISTILLATION: AUTOMATION OF MATERIAL FEED AND REMOVAL

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Automatic control of product, feed, and waste flow in the cryogenic distillation of carbon monoxide is essential for continuous operation of the still. A circuit to activate solenoid valves to the still to control the flow of materials has been designed, constructed, and tested. The unit is described in this report.

INTRODUCTION

Separation and enrichment of carbon-14 by cryogenic distillation of carbon monoxide is easily done. The recovery and enrichment of Hydro's carbon-14 wastes could reduce the long term disposal costs of these wastes and the resulting sale of the enriched carbon-14 could help fund this disposal /1/. Previous work in the Science Section on this distillation process has involved the calculation of the separation factors for the various isotopic combinations of carbon monoxide /2/, the construction of a prototype cryogenic still to test the performance of such a unit for isotope separation /3/, a computer model to simulate the distillation process /4/, and a description of an automatic unit to fill liquid nitrogen to the condenser of the still /5/.

The preliminary tests on the still have involved measurement of the number of theoretical plates as a function of the heat supplied to the reboiler, and as a function of the condenser temperature. (The condenser temperature controls the operating pressure of the still which in turn controls the boiling point of the liquid carbon monoxide.) These tests used natural carbon monoxide to separate carbon-13 monoxide from carbon-12 monoxide (carbon-13 is approximately 1% of the carbon-12 in natural samples). The tests at this stage of the experiments were performed under conditions of total reflux in which product, waste and feed flows are zero.

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The next step is to introduce feed, product, and waste flows to the still and automate that process. The basic operations are to admit a given quantity of gas to the still, while removing equivalent quantities of gas from the product and waste ends of the still. This report describes the unit that performs these operations.

DEFINITION OF THE CONTINUAL OPERATION PROCESS

Before the circuit can be described, it is necessary to discuss the actual operations that must take place when the still is run continually (i.e. with nonzero product, feed, and waste streams). In large scale distillation plants, the feed is usually added continuously with simultaneous removal of product and waste streams. In the isotopic enrichment experiments, the size of the feed stream is so small that it is difficult to meter it while still retaining any control over the volume of gas admitted to the still. Therefore, the continual method was chosen, in which plugs of gas could be regularly admitted and withdrawn. Larger quantities of gas are added at regular intervals and the total amount of gas either added or removed over an extended time period is the same as would be used in a continuous process (See Fig. 1). The continual method gives increased accuracy in flow monitoring and increases control over the material flows.

DESCRIPTION OF THE OPERATION

It will be useful to outline the philosophy of the operation of the unit and to define various terms used in the descriptions. Figure 1 shows a diagram of the layout of the valves in relation to the various parts of the still.

The volumes between the valves designated 1 and 2 are calibrated volumes and will be adjusted to feed and remove the correct volumes of gas. The top and bottom lines are normally evacuated and the gas exhausted; when radioactive samples are used, there is the provision to remove the gas in these lines through valve 4 to a suitable storage device. The middle line is filled from a carbon monoxide storage tank and provides the feed to the still.

Valves 3 and 5 are used to take occasional samples of the gas removed from the still. A sample container is attached to a port beneath valve 3 and is evacuated through valve 5. This is a semiautomatic procedure in that the operator throws a switch to open valve 5 when the sample chambers are to be prepared; this also suspends all other operations of the unit. Upon completion of the evacuation of the sample containers, a sample cycle can be selected, and the circuit automatically opens valve 3 at the appropriate time to admit gas to the sample container.

There are two clocks to control the timing of the cycle steps. The first clock controls the length of time that the various gas volumes are being evacuated or filled. This time period can be adjusted to any length to control the overall time that it takes the unit to complete one cycle (i e the waiting time between operations). Therefore the time on clock 1 may be much longer than that required to evacuate the gas lines (or fill the line from the feed tank). Clock 2 controls the amount of time that the gas lines are open to the still to withdraw gas from the still or admit feed. This time will be kept as short as possible, consistent with proper operation.

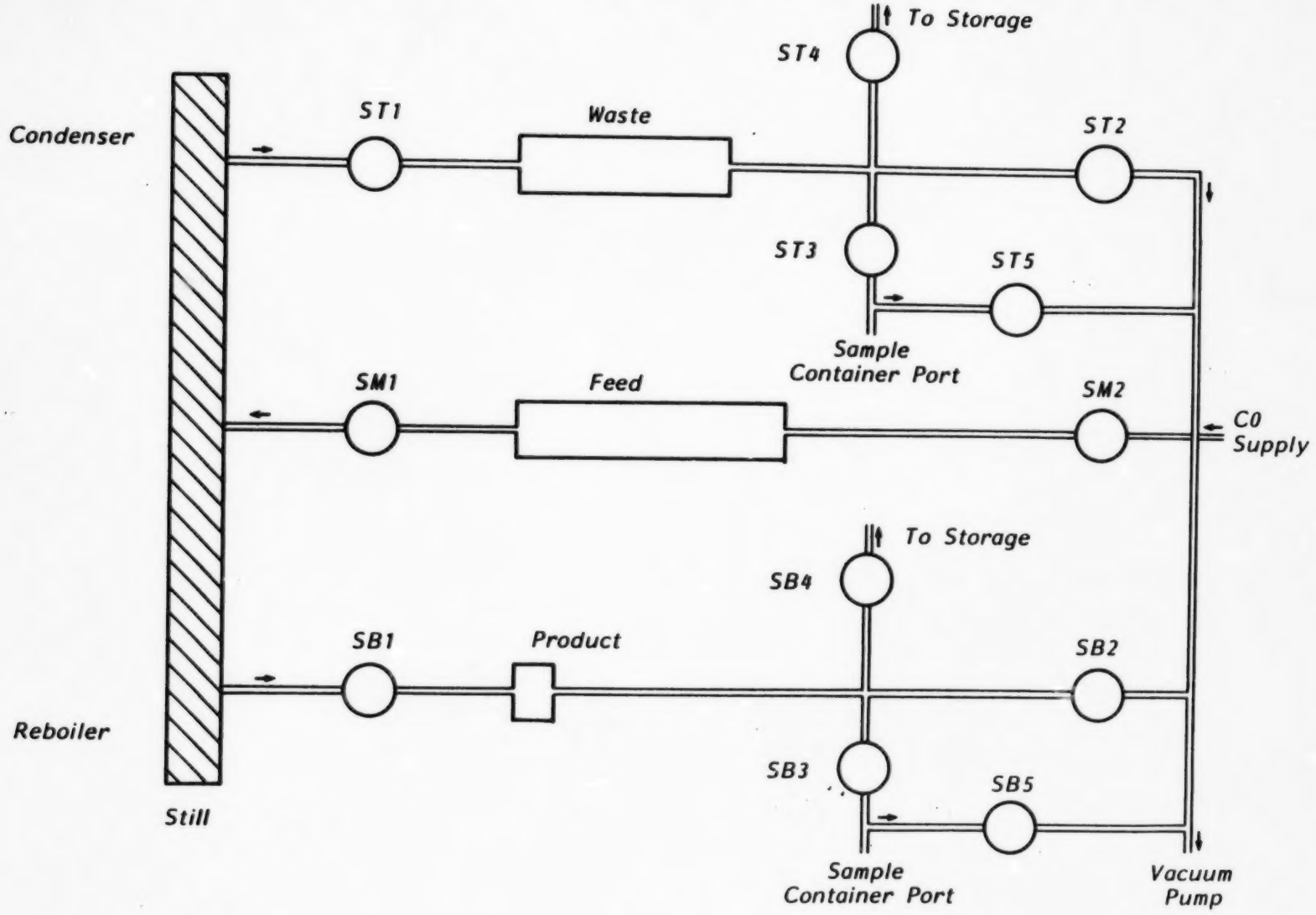

FIGURE 1

SCHEMATIC DIAGRAM OF THE LAYOUT OF
THE SOLENOID VALVES TO THE CRYOSTILL

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CIRCUIT DESCRIPTION

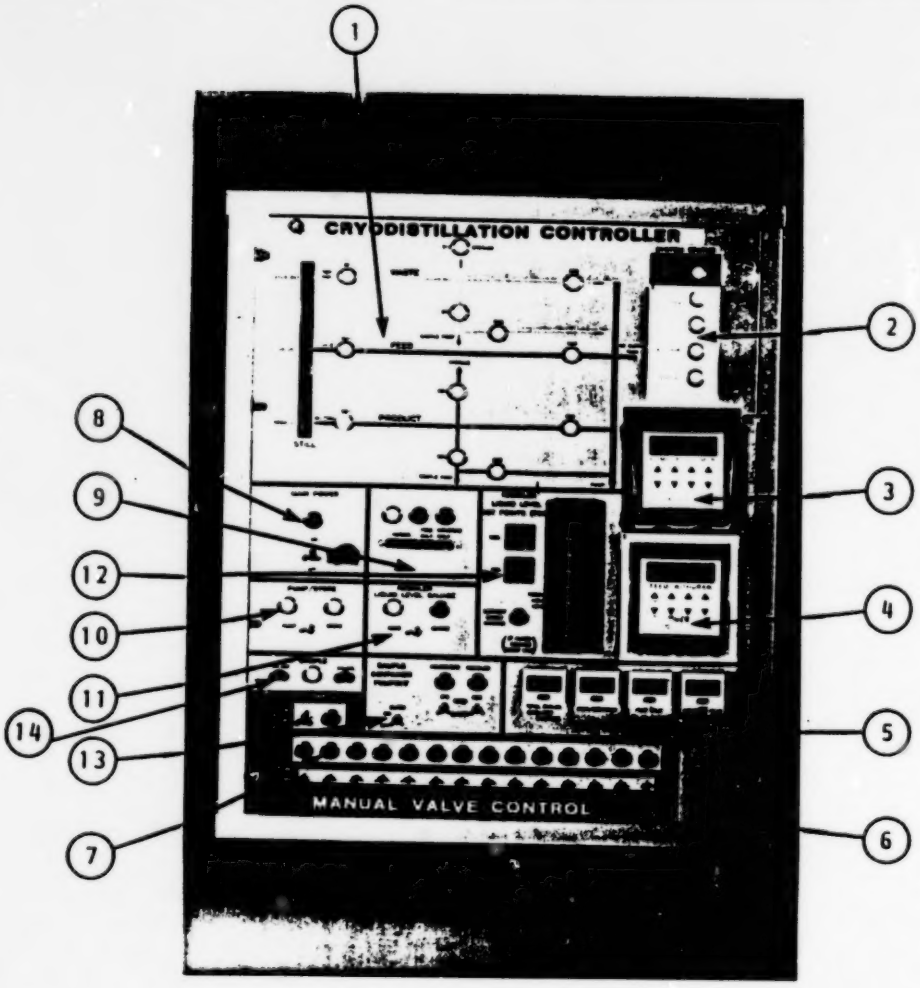
As stated above, the purpose of the unit is to control the flows of gases into and out of the still. The term "feed/withdraw" is used to describe this process in which feed gas is admitted to the middle of the column, while simultaneously product and waste gas are withdrawn from the bottom and top of the still. It is assumed that this is the normal sequence of operations. However, certain difficulties can arise. Specifically, the calibrated volumes used to feed and withdraw the gas are difficult to set accurately. Therefore, there is the possibility that the volumes will not be precisely matched and that after many hours of operation, the volume of liquid in the reboiler will increase or decrease from the initial volume, depending on whether too much or too little gas is fed into the still, relative to the withdrawn volumes. By measuring the volume of liquid carbon monoxide in the reboiler corrections can be made, and the cycles to correct the imbalance are called "Feed Only" or "Withdraw Only" depending on whether gas is fed into the still without product and waste being withdrawn (liquid level too low) or whether product and waste is removed without gas being fed into the still (liquid level too high). The operations "Feed only" and "Withdraw only" can be initiated by the circuit reading the liquid level and deciding the appropriate action or by having the operator select a switch on the front panel to initiate such cycles regardless of the liquid level in the reboiler. Generally any manual selection of operating conditions takes precedence over the automatic selection of the conditions.

Although the primary function of the unit is to control feed/withdraw cycles, there are other options available to the operator. The most important of these is the selection of the "sample mode" in which the sequence of operations is altered slightly to permit a sample of the product and waste streams to be removed for analysis. In this mode, the operator attaches a sample vial to the appropriate port on the gas handling system and requests a sample by pressing the appropriate button on the front panel. The unit then treats the next cycle as a feed/withdraw cycle regardless of other selections, and the cycle proceeds normally, with the exception that instead of immediately pumping the product and waste streams away, another valve opens for a short time to fill the sample vials with gas. The valve closes and the pumping begins as usual.

Other features of the unit are the override switches which allow all the valves to be manually operated. There are also sufficient combinations of switches to permit almost all semiautomatic operations; for example, the unit can be made to ignore the readings from the pressure gauge and perform fill/withdraw operations regardless of the volume of liquid in the still.

Figure 2 shows a photograph of the front panel of the control unit. The panel is roughly divided into three areas. The top and right hand side contain various indicator lamps and timers. The predominant feature is the schematic diagram of the still with lamps indicating the state of the solenoid valves. The timers giving the relative waiting times for the two parts of the cycle are on the right. The central portion contains the switches which allow the operator to select different conditions. The bottom section contains the manual override switches.

Details of the operation of this control unit and circuit diagrams are given elsewhere /6/.



1. *Valve Status*
2. *System Status*
3. *Timer 1*
4. *Timer 2*
5. *Cycle Counters*
6. *Reset*
7. *Override Switches*
8. *Main Power*
9. *Operation Select*
10. *Pump/Store Select*
11. *Liquid Level Gauge Select*
12. *Reboiler Liquid Level Set*
13. *Sample Container Pumpout*
14. *Sample Select*

FIGURE 2
FRONT PANEL PHOTOGRAPH

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SUMMARY

A unit to control the automatic operation of solenoid valves to admit feed and withdraw product and waste streams from the cryogenic still has been constructed and tested. A general description of the operation has been given here and further details are available in another report /6/.

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